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MOLECULAR STRUCTURE OF A NEW POTENTIAL PROPELLANT OXIDIZER 4,5-DINITROIMIDAZOLE (45DNI)

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A single crystal x-ray diffraction study verified that the target oxidizer molecule 4,5-dinitroimidazole (45DNI) had, indeed, been synthesized. Some structural features of 45DNI are compared with those of 1,4-dinitroimidazole (14DNI) and 2,4-dinitroimidazole (24DNI).

45DNI, in not planar, but the aromatic imidazole moiety is planar and the nitro groups twisted out the plane. The ring C-C and C-N bonds are all partial double bonds. 45DNI crystallizes with two crystallographically unique molecules in the monoclinic space group $P2_1/n$ (14) with unit-cell dimensions: a = 11.5360(8), b = 9.071(1), c = 11.822(1) Å, b = 107.640(6) deg, and Z = 8 and has a density of 1.781 g/cm^3. The molecular packing consists of infinite chains of 45DNI molecules hydrogen linked by two different hydrogen bonds [N(1)-H(1). . .N(31) and N(11)-H(11). . .N(3)] approximately in the diagonal ac direction (1 0 -1). The chains are held together by molecular interactions in the lateral directions.

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INTRODUCTION

The Propulsion Branch, in its quest for more energetic propellant oxidizers, initiated an investigation into heterocycles with high nitrogen and oxygen content. One group of heterocycles considered was the dinitro derivatives (DNI) of the five-membered ring imidazole (ref 1).

DNI is an energetic, dense, aromatic molecule which may have potential use in propellant formulations. However, this molecule can exist as several different isomers with respect to the positions of the substituted nitro groups. Substitution on the ring carbon atoms is preferable to substitution on the nitrogen atoms because molecules with nitro groups tend to be usually more stable than ones with nitramine groups. This limits the isomers of interest to 24DNI, 25DNI, and 45DNI. The structure of the 24DNI isomer was reported recently (ref 2).

This study was done primarily to confirm the identity of the product of a synthetic scheme employed by this laboratory (ref 3) to produce the target isomer 4,5-dinitroimidazole (45DNI). This target isomer is currently under consideration as a potential oxidizer substitute for RDX in solid and gel/slurry gun propellant formulations.

EXPERIMENTAL

A pale yellow crystal of 45DNI synthesized at this laboratory, having approximate dimensions of 0.20 x 0.20 x 0.25 mm, was mounted on a glass fiber. The preliminary examination and data collection were performed with Cu Kα x-radiation (λ = 1.54178 Å) on a Rigaku computer controlled diffractometer equipped with a graphite crystal incident-beam monochromater and a 12 kW rotating anode generator.

Cell constants presented in table 1 and orientation matrix for data collection were obtained at 23±1°C from least squares refinement using setting angles of 21 carefully centered reflections in the angular range of 79.57 deg < 2θ < 79.95 deg.

The intensity data were collected at a temperature of 23±1°C using the ω scan technique [θ scan width of 1.78 + 0.14 tan θ)] at a scan rate of 32.0 deg/min (in omega) to maximum 2 θ value of 120.0 deg . To assure good counting statistics, the intensities of weak reflections [I < 10.0σ (I)] were measured by accumulating the counts from four successive rescans. Stationary background counts were measured on each side of the reflection. The ratio of peak counting time to background was 2:1.

X-ray Data Reduction

A total of 3,783 reflections were measured, but only 1,897 were unique. The R_{int} value for averaged intensities was 0.055. As a check on system stability (electronic and crystal), the intensities of three representative reflections were measured after every 500 reflections. Over the course of data collection, the intensities of the standard reflections increased by 3.8%. To correct for this effect, a polynomial correction factor was applied to the data.

The reflections were corrected for Lorentz-polarization and secondary extinction effects (table 1). The secondary extinction coefficient applied was 3.82143E-05. Absorption corrections were not applied because azimuthal scan data of several reflections indicated that absorption was insignificant. The x-ray scattering factors were taken from Cromer and Waber (ref 4). Anomalous dispersion effects for heavy atoms were included in the structure factor calculations (ref 5) and the values of f' and f" were those of Creagh and McAuley (ref 6). On the basis of systematic absences, space group $P2_1/n$ was selected.

Structure Determination

Non-hydrogen atom positions were found by direct methods (ref 7) and the hydrogen atom positions were located by difference Fourier techniques. The hydrogen atoms were refined isotropically while the non-hydrogen atoms were refined anisotropically by full-matrix least-squares. The final cycle of full-matrix least-squares refinement, based on 1,390 reflections [$I > 3\sigma(I)$] and 216 variable parameters, converged with an unweighted agreement factor (R) and a weighted agreement factor (Rw) of 0.042 and 0.039, respectively.

Atomic coordinates and equivalent temperature factors, bond lengths, bond angles, and hydrogen bonding details are presented in tables 2 through 5, respectively. Two crystallographically unique molecules of 45DNI with their atom numbering schemes are shown in figure 1. The molecular packing viewed in three different unit-cell orientations is shown in figures 2 through 4. A space filled van der Waals diagram illustrating the hydrogen bond interaction is displayed in figure 5.

RESULTS AND DISCUSSION

45DNI crystallizes in the monoclinic primitive space group $P2_1/n$ with unit-cell dimensions: a = 11.5360(8) Å, b = 9.071(1) Å, c = 11.822(1) Å, and b = 107.640(6) deg. The asymmetric unit consists of two crystallographically unique 45DNI molecules, designated 45DNI(1) and 45DNI(2), which contain six carbon atoms, four hydrogen atoms, eight nitrogen atoms, and eight oxygen atoms. The symmetry operations of this space group generate eight molecules of 45DNI per unit cell which results in a calculated x-ray density of 1.781 g/cm^3.

Description of the 4,5-Dinitroimidazole Molecule

It is clearly shown in figure 1, that two nitro groups are substituted on the C(2) and C(4), and the C(21) and C(41) vicinal positions of the imidazole rings of the two respective molecules in this structure. This confirms that the target isomer 4,5-dinitroimidazole was made with the synthetic scheme devised by this laboratory.

4,5-dinitroimidazole, itself, is not a planar molecule but its ring moiety is planar. Similar conformational relationships also were reported for its 24DNI and 14DNI isomers (refs 2 and 8). The mean absolute deviation from the plane in the 45DNI(1) ring is 0.004 Å and is 0.003 Å in the 45DNI(2) ring. The greatest heavy ring atom deviation from planarity for both molecules is displayed by C(2), which is the carbon with the hydrogen atom substituent. In 45DNI(1), C(2) deviates from the plane of its ring by -0.008(3) Å and in 45DN(2) C(21) deviates by 0.006(3) Å. All hydrogen atoms are also displaced from the plane of the ring. In 45DNI(1), H(1) which is bonded to N(1) is off the plane by 0.067 Å and H(2) which is bonded to C(2) is off the plane by 0.018 Å. In 45DNI(2), its counterparts H(11) and H(21) are off the plane by 0.152 and 0.078 Å, respectively.

In each molecule, both nitro groups are twisted out of the ring plane and each twist axis is also displaced from the plane. The 4-nitro group in 45DNI(1) is twisted out of the ring plane by about 28 deg and the 5-nitro group is twisted out by 18 deg. N(4) in the 4-nitro group is displaced from the ring plane by 0.058 Å while N(5) of the 5-nitro group is off the plane by -0.117 Å. In 45DNI(2), the 4-nitro group is twisted out of the ring plane by 17 deg and the 5-nitro group is twisted out by 172 deg. In 45DI(2), the nitrogen atom N(41) of the 4-nitro group is displaced 0.028 Å and N(51) in the 5-nitro group is displaced 0.117 Å.

In the 14DNI isomer, the 1-nitro group is twisted 9 deg and the 4-nitro group is twisted 2 deg out of the ring plane. In 24DNI, the 2-nitro group is twisted about 1 deg and the 4-nitro group about 8 deg. In general, the nitro group twist angles for the 45DNI isomer are larger than either those of the 24 or 14 isomers. This is not unexpected because the vicinal nitro group conformation in the 45 isomer should cause greater stearic hinderance or molecular interactions between nitro groups than do the non-vicinal nitro conformations in the other isomers.

The large adjacent angles between the ring and the two nitro groups for both 45DNI(1) and 45DNI(2) [N(5)-C(5)-C(4) is 133.8(2) deg, N(4)-C(4)-C(5) is 130.0(2) deg, and N(51)-C(51)-C(41) is 136.1(3) deg, N(41)-C-(41)-C(51) is 131.0(2) deg] is further evidence of intramolecular repulsion between the juxtaposed nitro groups.

The bond lengths (table 2) for C(4)-C(5) and C(41)-C(51) are 1.355(3) and 1.357(4) Å, respectively. These are partial C-C double bonds because these interatomic distances are closer to the accepted double-bond length of 1.337(4) Å than to the single-bond length of 1.541(3) Å (ref 9). The C-C bond lengths of the 45DNI isomer agree with C(4)-C(5) reported for 14DNI isomer with a value of 1.354(3) Å, but are shorter than observed in the 24DNI isomer [C(4)-C(5) 1.370 Å].

All four ring C-N bonds (table 2) in this isomer are also significantly shorter than the accepted C-N single bond length of 1.472(5) Å. Hence, each C-N bond is a partial double bond rather than a single-bond. Since all the ring bonds are partial double bonds, 45DNI may be considered an aromatic molecule.

In 24DNI, C(2)-N(3) is the shortest C-N bond in the ring with a value of 1.265(9) Å, while its neighboring C(2)-N(1) bond is the longest C-N bond (1.363 °) in the ring. In 45DNI, C(21)-N(31) in 45DNI(2) is the also the shortest C-N bond with a value of 1.316(4) Å, but its neighboring C(21)-N(11) bond has a value of 1.346(4) Å. However, C(21)-N(11) is shorter than its remaining C-N ring bonds. In 45DNI(1), the analogous C(2)-N(3) bond has a value of 1.326(3) Å but is now equivalent with its neighboring C(2)-N(1) with a value of 1.329(4) Å.

All N-O bond lengths in the 45DNI nitro groups are in the range of accepted single bonds [1.14 to -1.21(14) Å].

Description of the Crystal Structure

The crystal structure is composed of a network of infinite chains of 45DNI molecules linked by zigzag hydrogen bonds oriented approximately along (1 0 -1), the ac direction. The dihedral angle between the planes of imidazole moieties in adjacent 45DNI molecules within the chain is 40.52 deg. The chains are held together by lateral intermolecular forces. This molecular packing is demonstrated in different unit-cell orientations in figures 1 through 4.

Each molecule has one hydrogen atom and one acceptor nitrogen atom participating in two unique hydrogen bonds. The data in table 4 indicate that N(11)-H(11)...N(3) is the stronger hydrogen bond because it has an almost linear bond angle of 172(3) deg and relatively short interatomic distances [N(11)..N(3) 2.848(3) and H(11)..N(3) 1.98(5) Å] . The weaker bond, N(1)-H(l)...N(31), has a bond angle of 152(4) deg and longer interatomic distances [N(1)...N(31) 2.865(4) and H(1)..N(31) 2.07(5) Å]. The hydrogen bond interaction is depicted in figure 5 where the overlapping of the nitrogen and hydrogen van der Waals atomic radii is clearly shown.

The crystalline forces manifested by this molecular packing affect the structure of the individual molecules. This is demonstrated by the observed differences in the analogous molecular parameters of the two crystallographically unique 45DNI

molecules. Furthermore, these crystalline forces can also perturb the molecular structure to such an extent that the molecular parameters may differ significantly from those values calculated by quantum mechanics for an isolated molecule. Work is currently in progress at this laboratory to calculate these parameters by ab initio and semi-empirical methods (ref 10).

CONCLUSIONS

The target molecule, the 4,5-dinitroimidazole isomer, was selectively synthesized by this laboratory.

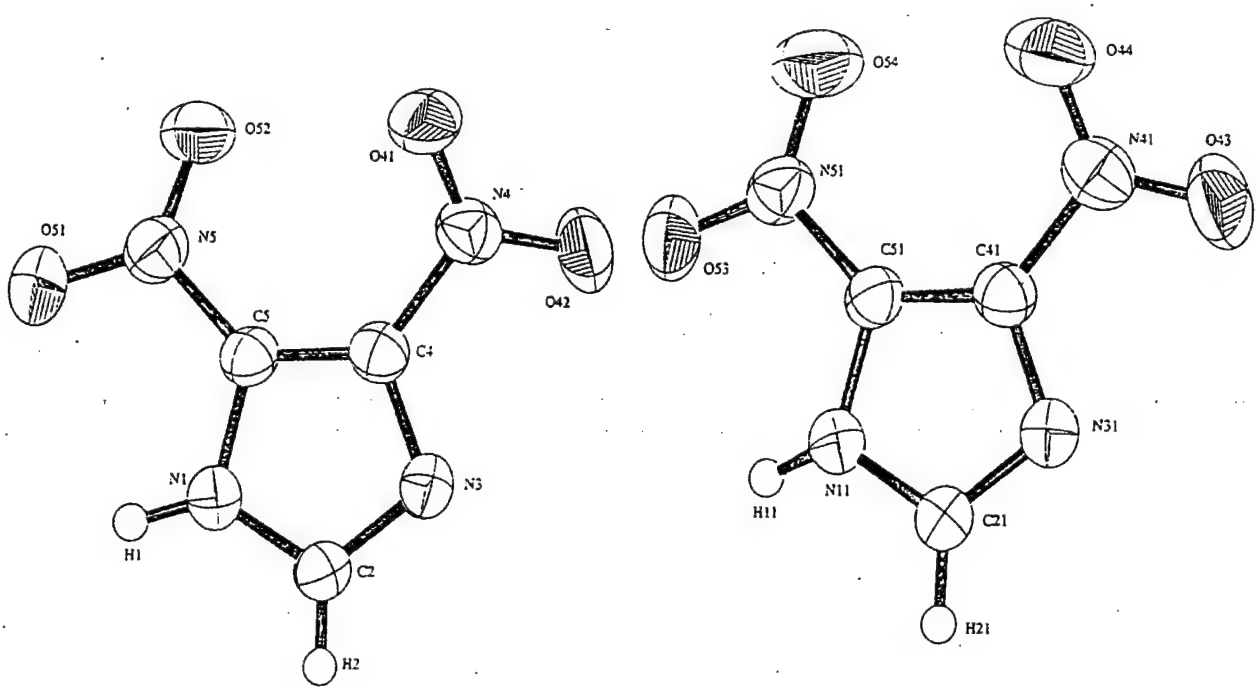
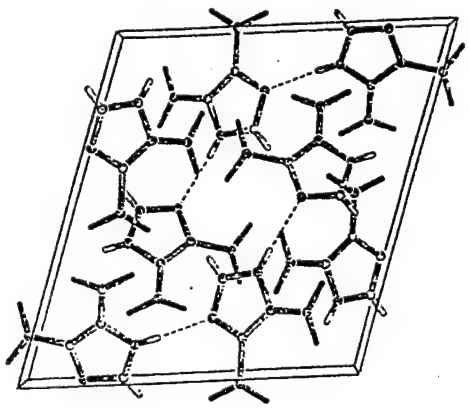
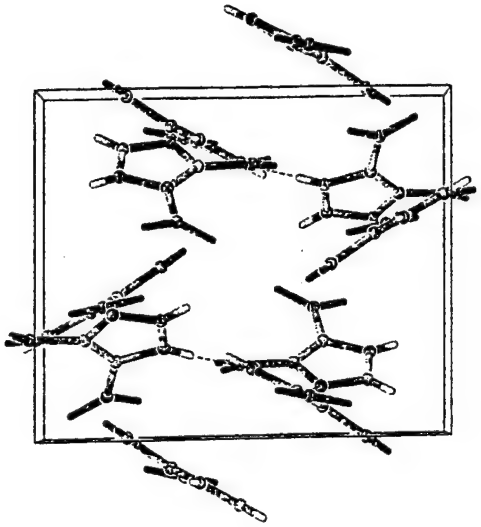


Figure 1
45DNI molecular conformations and atom numbering scheme



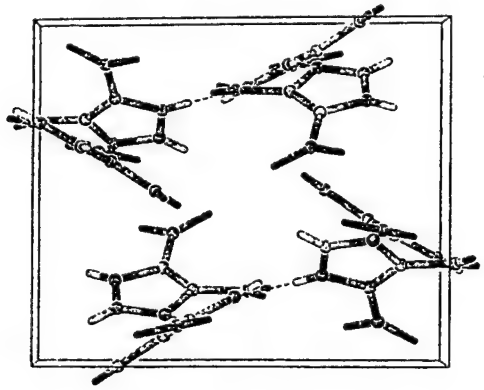
Hydrogen bonds are shown as dashed lines

Figure 2
Unit-cell diagram viewed down the b axis



Hydrogen bonds are shown as dashed lines

Figure 3
Unit-cell diagram viewed down the a axis



hydrogen bonds are shown as dashed lines

Figure 4
Unit-cell diagram viewed down the c axis

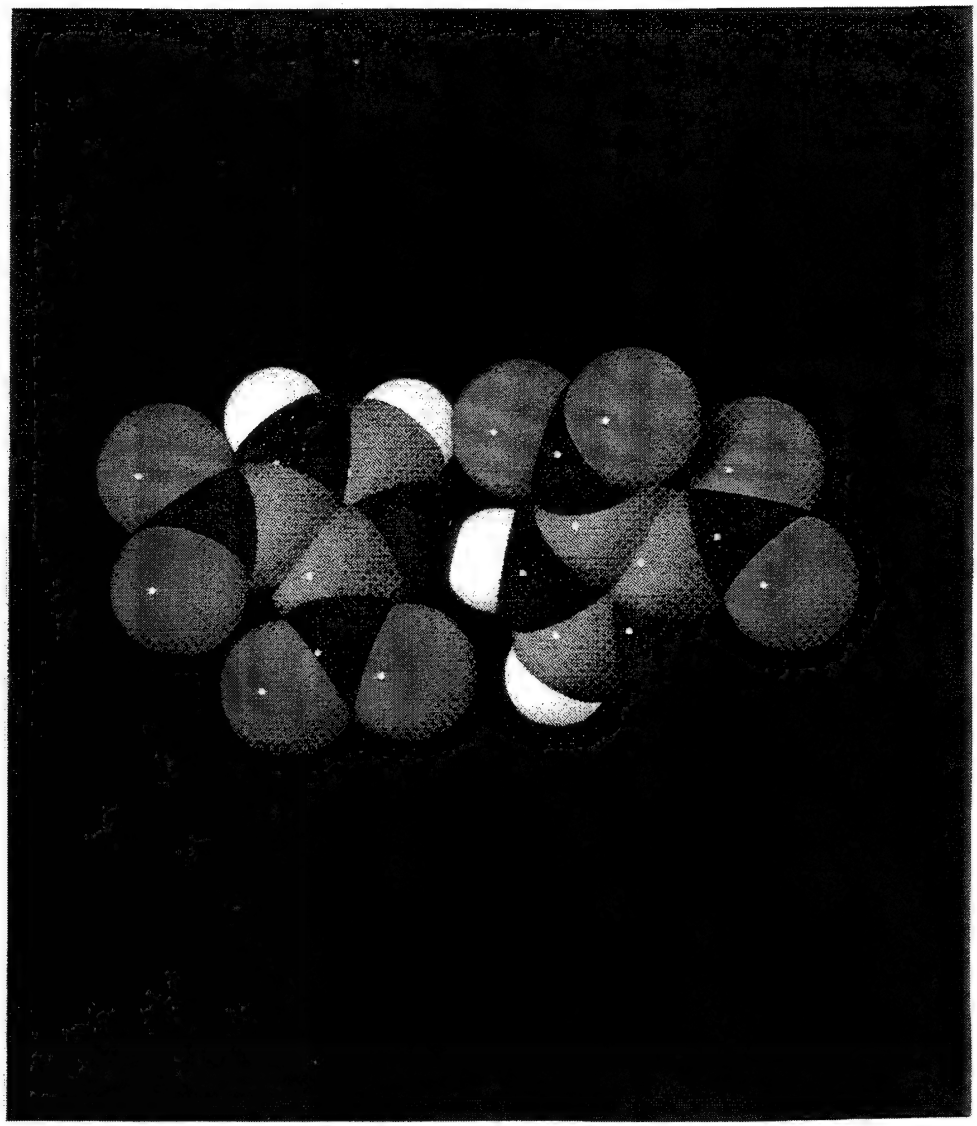


Figure 5
45DNI molecular configuration using space filled van der Waals atomic radii

Table 1
45DNI crystal data collected at 23±1°C*

Molecular formula	C3H2N4O4
Formula weight	158.07
Crystal System	Monoclinic
Space group	$P2_1/n$ (#14)
a	11.5360(8) Å
b	9.071(1) Å
c	11.822(1) Å
β	107.640(6) deg
V	1178.9(2) Å^3
Z	8
D	1.781 g/cm^3
λ (Cu Kα)	1.54178 Å
u	14.83 cm^{-1}
F(000)	640
Scan type	ω
2θ max	120.2 deg
Total reflections	3783
Unique reflections	1807
No. reflections [I > 3.σ (I)]	1390 (R_{int} = 0.055)
h	0 to 12
k	-2 to 10
ℓ	-13 to 12
Secondary extinction	3.8213E-05
R; Rw	0.042; 0.039
GOF	2.21
Final Diff Fourier	
Max peak	0.24 e-/Å^3
Min peak	-0.24 e-/Å^3

* X-ray diffraction data collected by Molecular Structure Corporation, The Woodlands, Texas.

Table 2
45DNI atomic coordinates and equivalent thermal parameters and their esd's in parentheses

Atom	x/a	y/b	z/c	Beq
45DNI(1)				
0(41)	1.0736(1)	0.2161(2}	0.7451(2)	4.66(5)
0(42)	1.0574(2)	0.1947(2)	0.5600(2)	5.40(6)
0(51)	0.7483(2)	0.3904(2)	0.8119(2)	4.97(5)
0(52)	0.9304(1)	0.4330(2)	0.8042(2)	4.46(5)
N(1)	0.6948(2)	0.2381(2)	0.6063(2)	3.08(5)
N(3)	0.8158(2)	0.1417(2)	0.5130(2}	3.17(5)
N(4)	1.0140(2)	0.2098(2)	0.6414(3)	3.46(5)
N(5)	0.8322(6)	0.3712(2)	0.7696(2)	3.45(5)
C(2)	0.7021(2)	0.1590(3)	0.5140(3)	3.21(6)
C(4)	0.8826(2)	0.2126(2)	0.6118(3)	2.81(6)
C(5)	0.8105(2)	0.2738(2)	0.6707(3)	2.78(6)
H(1)	0.635(3)	0.261(3)	0.634(3)	5.2(7)
H(2)	0.634(6)	0.124(3)	0.461(3)	4.2(6)
45DNI(2)				
0(43)	0.9318(2)	0.1357(2)	-0.0569(2)	5.72(6)
0(44)	0.7448(2)	0.0880(2)	-0.0714(2)	5.78(6)
0(53)	0.6845(2)	-0.0212(2)	0.2731(2)	5.19(6)
0(54)	0.6540{2)	-0.0507(3)	0.0849(2)	6.58(7}
N(11)	0.8850(2)	0.1303(2)	0.3000(3)	3.09(5)
N(31)	0.9832(2)	0.1968(2)	0.1740(3)	3.43(6)
N(41)	0.8491(2)	0.1155(2)	-0.0142(2)	3.87(6)
N(51)	0.7093(2)	-0.0002(2)	0.1807(2)	3.70(6)
C(21)	0.9833(2)	0.1982(3)	0.2583(3)	3.50(7)
C(41)	0.8786(2)	0.1296(2)	0.1137(3)	3.02(6)
C(51)	0.8166(2)	0.0871(2)	0.1891(3)	2.81(5)
H(11)	0.876(3)	0.104(5)	0.367(5)	10(1)
H(21)	1.046(2)	0.229(3)	0.356(2)	4.1(6)

Table 3
45DNI bond lengths in angstroms (Å) and their esd's in parentheses

Atom	Atom	Distance	Atom	Atom	Distance
0(41)	N(4)	1.212(3)	0(42)	N(4)	1.220(3)
0(43)	N(41)	1.221(3)	0(44)	N(41)	1.214(3)
0(51)	N(5)	1.229(2)	0(52)	N(5)	1.219(2)
0(53)	N(51)	1.224(3)	0(54)	N(51)	1.209(4)
N(1)	C(2)	1.329(4)	N(1)	C(5)	1.360(3)
N(1)	H(1)	0.87(3)	N(3)	C(2)	1.326(3)
N(3)	C(4)	1.352(4)	N(4)	C(4)	1.449(3)
N(5)	C(5)	1.426(3)	N(11)	C(21)	1.346(3)
N(11)	C(51)	1.366(4)	N(11)	H(11)	0.86(5)
N(31)	C(21)	1.316(4)	N(31)	C(41)	1.355(3)
N(41)	C(41)	1.452(4)	N(51)	C(51)	1.447(3)
C(2)	H(2)	0.90(3)	C(4)	C(5)	1.355(3)
C(21)	H(21)	0.97(3)	C(41)	C(51)	1.357(4)

Table 4
45DNI bond angles in degrees and their esd's in parentheses

Atom	Atom	Atom	Angle	Atom	Atom	Atom	Angle
C(2)	N(1)	C(5)	107.0(2)	C(2)	N(1)	H(1)	133(2)
C(5)	N(1)	H(1)	119(2)	C(2)	N(3)	C(4)	104.0(2)
0(41)	N(4)	0(42)	124.1(2)	0(41)	N(4)	C(4)	118.4(3)
0(42)	N(4)	C(4)	117.4(3)	0(51)	N(5)	0(52)	124.4(2)
0(51)	N(5)	C(5)	117.2(2)	0(52)	N(5)	C(5)	118.4(2)
C(21)	N(11)	C(51)	105.8(3)	C(21)	N(11)	H(11)	126(2)
C(51)	N(11)	H(11)	127(2)	C(21)	N(31)	C(41)	104.7(2)
0(43)	N(41)	0(44)	124.6(3)	0(43)	N(41)	C(41)	116.8(2)
0(44)	N(41)	C(41)	118.5(2)	0(53)	N(51)	0(54)	125.2(2)
0(53)	N(51)	C(51)	116.8(3)	0(54)	N(51)	C(51)	118.0(3)
N(1)	C(2)	N(3)	112.4(2)	N(1)	C(2)	H(2)	120(1)
N(3)	C(2)	H(2)	126(1)	N(3)	C(4)	N(4)	118.7(2)
N(3)	C(4)	C(5)	111.2(2)	N(4)	C(4)	C(5)	130.0(3)
N(1)	C(5)	N(5)	120.4(2)	N(1)	C(5)	C(4)	105.4(2)
N(5)	C(5)	C(4)	133.8(2)	N(11)	C(21)	N(31)	112.7(3)
N(11)	C(21)	H(21)	117(1)	N(31)	C(21)	H(21)	129(1)
N(31)	C(41)	N(41)	118.6(3)	N(31)	C(41)	C(51)	110.4(3)
N(41)	C(41)	C(51)	131.0(2)	N(11)	C(51)	N(51)	117.2(2)
N(11)	C(51)	C(41)	106.4(2)	N(51)	C(51)	C(41)	136.1(3)

Table 5
4,5-DNI hydrogen bond distances in angstroms (Å) and angles in degrees and their esd's in parentheses

D-H..A*	D-H	H. . .A	D. . .A	Angle
N(1)-H(1)..N(31)	0.87(5)	2.07(5)	2.865(7)	152(4)
N(11)-H(11)..N(3)	0.86(3)	1.98(5)	2.848(3)	172(3)

* D is the hydrogen donor atom and A is the hydrogen acceptor atom.

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